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CHARACTERIZATION OF CERAMIC COMPOSITE MATERIALS USING TERAHERTZ NON-DESTRUCTIVE EVALUATION TECHNIQUES (PREPRINT)

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14. ABSTRACT
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Characterization of Ceramic Composite Materials Using Terahertz Non-Destructive Evaluation Techniques

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Abstract: THz time-domain reflection imaging is utilized as non-destructive evaluation technique for ceramic composite materials in order to characterize changes in material properties due to mechanical and thermal strain effects.
OCIS codes: (110.6795) Terahertz imaging; (300.6495) Spectroscopy, terahertz

1. Introduction

The use of non-destructive evaluation (NDE) techniques to characterize defects such as rust, voids, etc. in materials and to analyze and predict strain and stress induced breakdown. The use of terahertz (THz) imaging as an evaluation tool for novel materials and systems has found much success in recent years [1-3]. While suggested as a potential NDE tool for use in the field of ceramic and ceramic matrix composite materials, the use of THz spectroscopy and imaging in the examination of the effects of mechanical and thermally induced strain on ceramic composite materials is not well established. In order to assess the effectiveness of THz imaging for use in the analysis of ceramic composite material health, it is necessary to determine if THz spectroscopic imaging can clearly highlight areas of the samples that have been affected by mechanical and thermal stress.

Ceramic matrix composite materials are the ideal choice for the next generation of thermal protection systems due to their high strength and thermal resistance. The first round of data acquisition consisted of spectroscopic imaging of all of the samples yet to receive any heat or mechanical fatigue treatment. Subsequent rounds consisted of sample imaging following treatments of either thermally or mechanically induced stress of varying magnitudes. Combinations of the two differing types of stress have also been studied. Comparison of the data both in the time-domain and frequency-domain acquired from imaging of the untreated and treated samples assesses whether or not the magnitude and extent of stress-induced changes can be monitored using terahertz imaging and spectroscopy.

THz time-domain images were acquired using a commercial system manufactured by Teraview. Ultrafast laser pulses with an 800 nm center wavelength and 100 fs pulse width triggered a fiber-coupled GaAs photoconductive antenna (PCA). Collimated THz light from the PC antenna transmitter was focused via a 50 mm focal length lens (f# = 2) onto the samples at a near-normal incident angle. The reflected radiation was detected by a PCA receiver module, based on LT-GaAs, with an identical lens configuration. When the system is optimized and calibrated using a metal reference reflection target, the typical bandwidth of the detected THz pulse exceeds 3 THz.

2. Reflection Imaging

Tests were conducted on both oxide-based and silicon nitride carbon based CMC samples. Future work will also include silicon carbide-silicon nitride carbon (SiC-SiNC) based samples. A 5.9 x 17.4 cm area is scanned, containing both an aluminum reference and the CMC sample. A full time-domain waveform, 250 ps long, is acquired for each 0.5 x 0.5 mm pixel. The samples of dimension 15.7 x 1.3 x 0.2 cm are segmented into 11 blocks of equal area. This allows for comparative measurements to be isolated to the same area of the sample. Certain treatments are only applied to a specific area of the sample and analysis is focused on those sections rather than on the entire sample.

Fig. 1 shows a THz image comparing the reflection from several types of SiNC samples. Note that the textured weave on the surface of the sample is clearly visible. The heat treated samples appear to have a different surface roughness than the undamaged baseline or fatigue treated samples. In the oxide samples, however, no change in surface roughness is visibly apparent following any type of treatment. Quantitative analysis of this surface roughness phenomena is underway.

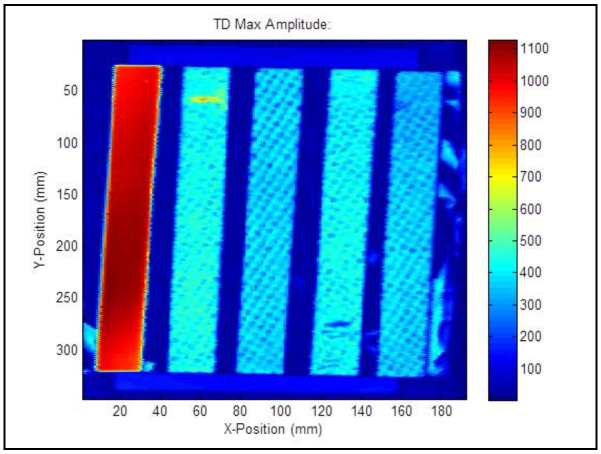


Fig. 1 Spectroscopic Image of SiNC samples based on the maximum time-domain amplitude.
Samples include, from left to right: Aluminum Reference, Baseline, Heat Treated 100 hrs, Fatigue Treated, Heat Treated 10 hrs.

3. Results

In order to analyze the acquired data, a program was written in MATLAB that generates images based on the maximum amplitude of the time-domain pulse as well as changes in the arrival time of the pulse (time of flight). Images are also generated based on the electric field amplitude at specified frequencies and frequency weighted reflectivity. In this latter case, the amplitude of each pixel is based on the integration of the full spectral content of the detected pulse. Graphs of normalized reflectivity (for a specific block) corrected with the Rayleigh roughness factor are plotted as a function of frequency. In all cases, the reflectivity is based on comparison of a THz pulse reflected from a CMC sample compared to the THz pulse reflected from the aluminum reference.

We also examine changes in the reflectivity of these samples pre- and post- stress treatment over smaller spatial sub-domains. A grid system has been developed that allows for point-by-point analysis of the imaging data which makes it possible to compare specific spatial points, with a spatial resolution of less than 1 mm, on samples prior to and following stress treatment. This analysis approach assesses the spatial variance of any stress induced changes in the THz reflectivity. In addition, in depth-statistical analysis on the reflectivity data is currently underway which will be used to aid in determining if the data overlapped in error bars is distinguishable.

Fig. 2(a) shows the reflectivity as a function of frequency for a SiNC sample before and after undergoing a heat treatment. The oscillatory trend from 0-600GHz is evident in all SiNC samples and is believed to be a characteristic trait of these materials. Following heat treatment of 1200 deg. C for 100 hours, the magnitude of the reflectivity maintained the same general trend of the baseline with a decrease in reflectivity by approximately 0.1 for the entire frequency span shown. Fig. 2(b) is a plot of the reflectivity as a function of frequency for a SiNC sample before and

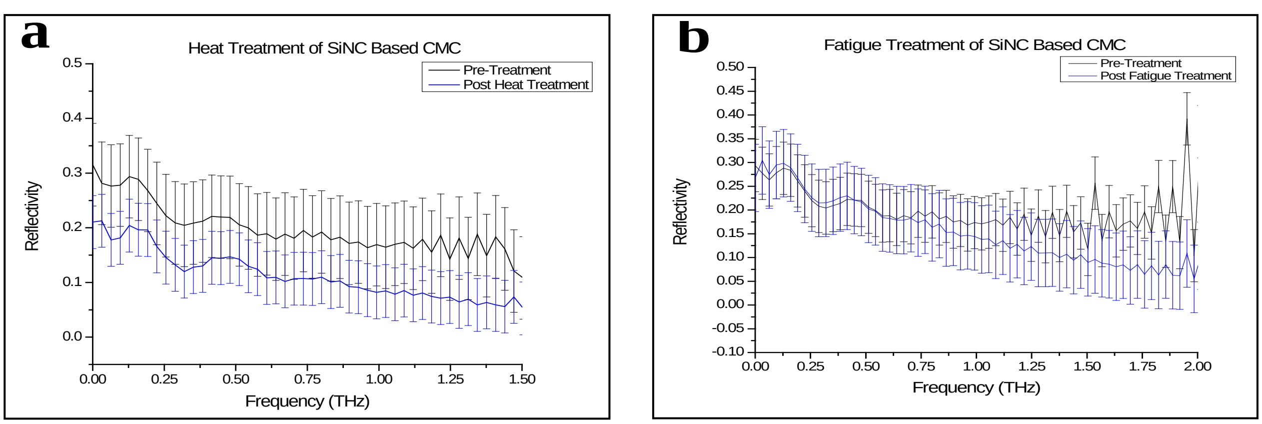
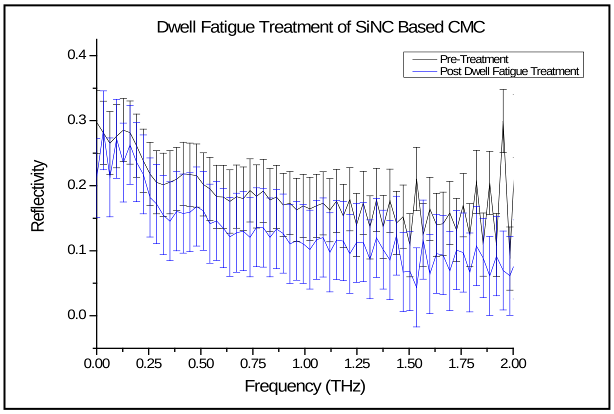


Fig. 2 (a) SiNC based sample prior to treatment (black) and following a heat treatment for 100 hours at 1200 deg. C (blue). (b) SiNC based sample prior to treatment (black) and following a fatigue treatment (blue).



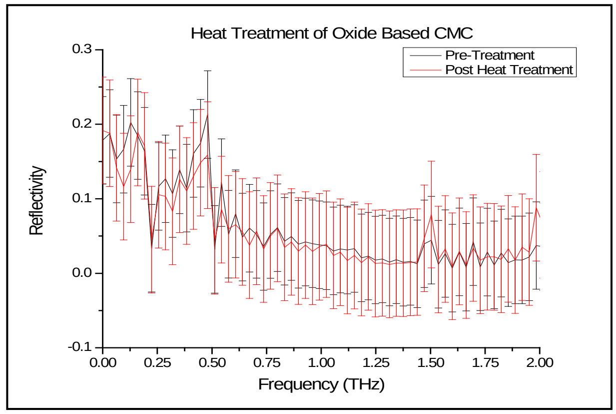


Fig. 3 (a) Dwell Fatigue Treatment on SiNC sample. (b) Dwell Fatigue Treatment on an Oxide sample. In both figures, the baseline measurement is indicated by the black line.

after undergoing a fatigue treatment. In the lower frequencies, out to 600GHz there is no apparent change in the magnitude of reflectivity. However, at higher frequencies, the reflectivity diverges, with the fatigue treated sample exhibiting a lower reflectivity.

Dwell fatigue is a simultaneous combination of fatigue and heat treatment induced simultaneously and are concentrated only on the middle of the sample. Sample reflectivity following a dwell fatigue treatment is represented in Fig. 3(a). Unlike the isolated fatigue treatment in lower frequencies the magnitude of reflectivity is less than that of the baseline. There is also no divergence at the higher frequencies. Due to the evidence that fatigue treatments have no effect on reflectivity at these frequencies, the fatigue contribution to the dwell treatment is negligible. For the range of 0.25-2.0 THz the magnitude of change in reflectivity is ~0.1, the same as an isolated heat treated sample. This consistent decrease in reflectivity from the dwell fatigue treated sample leads to the conclusion that the prominent contributing factor is the heat portion. Spectral imaging of the oxide based CMC materials also shows a downward trend in reflectivity for higher frequencies which is similar to that seen in the SiNC samples. The fluctuating reflectivity features seen between 0-0.25THz along with the spike in reflectivity at 1.6 THz are characteristic among the oxide CMC samples. Fig 3(b) shows measurements from an oxide sample before and after receiving heat treatment at 1000 deg. C for 100 hrs. The trend and magnitude of reflectivity do not change following heat treatment, as seen in the figure. Further, both fatigue treatments and dwell fatigue treatments also have no effect on reflectivity, as no measureable change was measured.

4. Conclusion

Using Terahertz reflection imaging, the characterization of ceramic matrix composite materials has been established. As a result of comparative reflectivity calculations for samples pre- and post-treatment, there is no distinguishable difference between reflectivity values in this frequency band for oxide based samples. However, SiNC based samples demonstrate changes in reflectivity following heat treatments, but not fatigue.

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